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Photoelectron Emission Spectroscopy of Liquid Water.

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Paul Delahay and Kathrin von Burg

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PHOTOELECTRON EMISSION SPECTROSCOPY OF LIQUID WATER

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The threshold energy $E_t = 10.06$ eV (0.002 eV standard deviation) is determined for photoelectron emission by liquid water and is correlated with $E_t = 8.45$ eV for $\text{OH}^-(\text{aq})$. Free energy changes and standard reduction potentials are calculated for both emission processes. Reorganization free energies are correlated to solvation free energies for $\text{H}_2\text{O}^+(\text{aq})$ and $\text{OH}^-(\text{aq})$.

The photoionization of liquid water was investigated in [1] by means of photoelectron emission spectroscopy, and the emission threshold energy was estimated at $E_t = 9.3 \pm 0.3$ eV. The same photon energy (within ± 0.1 eV) was deduced for the injection of holes in liquid water in a photoelectrochemical study of gold electrodes [2]. Great care was taken in [1] to avoid spurious emission by traces of insoluble organic impurities. Yet, subsequent work on inorganic anions (see fig. 1 in [3]) in which two potential sources of contamination were eliminated (sec. 1) indicated that the 9.3 eV threshold energy is definitely too low. A new investigation of water was undertaken. Its results are reported here and are correlated with the data for photoelectron emission by hydroxide ion.

1. Results

The emission spectrometer was the same as in [1] except for two improvements. The polyethylene cylinder insulating electrically the rotating quartz disk (target) from its metallic shaft was replaced by a nylon cylinder. The quartz disk was fastened to the insulating cylinder by means of a nylon screw instead of being cemented. These changes were made prior to the work in [3,4]. The LiF window between the hydrogen lamp and the monochromator was replaced by a new window after the determination of only three spectra. This was done to lower the noise level and stray light errors arising from fogging of the window by color centers. Yields were corrected for attenuation of the photon flux by water vapor in the 1 mm gap between the water surface being irradiated and the opposite LiF window. The correcting factor computed from data in [5] was taken to decrease linearly from 1.1 to 1.05 from 9.90 to 10.77 eV. The signal of the photomultiplier tube monitoring the photon flux was multiplied by a factor increasing linearly by 8% between 9.90 and 10.77 eV (data from [6]). This correction compensated for attenuation of the photon flux in the LiF window opposite to the water surface. Spectra were determined repeatedly until a constant threshold energy was obtained.

The spectrum of fig. 1 shows no noticeable emission below 9.9 eV (vs. ca. 9.3 eV in [1]), and yields are markedly lower than in [1], e.g., by a factor of ca. 6 at 10.3 eV. Each point of the spectrum in fig. 1 represents the average of eight measurements each of which was made with a fresh water sample. The threshold energy $E_t = 10.06$ eV (0.002 eV standard deviation) was determined from a linear plot (97 points from 10.2 to 10.77 eV, fig. 1) of the yield Y to the power 0.5 against photon energy [7]. A statistical F-test [8] unambiguously shows that the best linear fit of the data is obtained with the exponent 0.5 of the yield. Departure from linearity near the threshold,

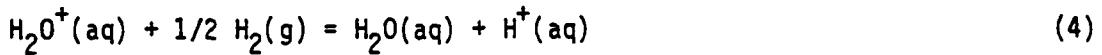
which is usual [3,4], arises from complications not taken into account in theory [7]. Logarithmic dependence of the yield on photon energy fits the data very poorly ($F \approx 2,000$), and there is no Urbach edge (cf. [1]).

2. Photoelectron emission by liquid water

Emission by liquid water will be interpreted on the basis of the equation [3,4],

$$\Delta G_e = \Delta G + \Delta G_H - \Delta G_r + |e|x, \quad (1)$$

where e is the electronic charge and x the surface potential at the water-water vapor interface. The free energies ΔG_e , ΔG_r , $-\Delta G$, $-\Delta G_H$ pertain, respectively, to the reactions,



The free energy of photoelectron emission ΔG_e according to (2) includes the contribution for electronic polarization of the medium by $H_2O^+(aq_*)$ but not for orientation polarization (Franck-Condon principle). The reorganization free energy ΔG_r for (3) is the sum of the contributions for orientation polarization of the medium and vibrational and rotational relaxation of the ion produced by photoionization. The latter contribution is negligible since interatomic distances hardly change in the photoionization of H_2O in the gas phase [9]. The free energy $\Delta G_H = 4.50$ eV [10] correlates the free energies referred to the vacuum level of the electron to the free energies referred to the $H^+(aq)/1/2 H_2(g)$ couple. The quantity ΔG_H does not include the contribution from the surface potential $x = \pm 0.1$ V [11].

The free energy ΔG_e differs from the threshold energy E_t because of the contribution from the surface potential x and possibly a systematic shift

inherent to the extrapolation method and theory [7] yielding E_t . It was found in [3] that measured E_t 's and calculated ΔG_e 's for Cl^- , Br^- , I^- agree within the uncertainty arising from the surface potential. There is no reason to expect a different conclusion for other photoionized species, and the relationship $\Delta G_e \approx E_t$ should hold in general. Thus, we set $\Delta G_e \approx E_t = 10.06$ eV in (1) for liquid water.

Lower and upper limits of the free energy ΔG in (1) will be obtained. The ions H_2O^+ (aq) and OH^- (aq) are of comparable size, and consequently their solvation and reorganization free energies should not be very different. If one uses the value $\Delta G_r = -1.98$ eV for OH^- (aq) (sec. 3) in (1), one computes $\Delta G = 3.58$ eV. Another limit can be set on the value of ΔG by using the gas-phase first ionization energy of water $I = 12.61$ eV [9]. The quantity I is an enthalpy, and the entropy contribution to gas-phase ionization is neglected. One has $I > \Delta G_e$ for two reasons: electronic polarization of the medium by the ion H_2O^+ (aq_{*}) produced by (2) and distribution of density of states in the liquid [12]. One can assume that the free energy of vaporization of water is nearly compensated by the contribution to the solvation free energy of $\text{H}_2\text{O}^+(g)$ not arising from the ionic charge. Thus, $I - \Delta G_e > -\Delta G_{ep}$, where ΔG_{ep} is the contribution from electronic polarization of the medium to the solvation free energy ΔG_s of H_2O^+ (aq). The quantity ΔG_s is the solvation real energy in electrochemical terminology (e.g., [13]). Thus, $\Delta G_{ep} > 10.06 - 12.61$ eV or $\Delta G_{ep} > -2.55$ eV. The ratio $\Delta G_r/\Delta G_s$ is close to 0.50 for the halides (Table 1) and OH^- (aq) (sec. 3), and it will be assumed that this ratio is also near 0.50 for H_2O^+ (aq). Hence, $\Delta G_{ep} \approx \Delta G_r$ for H_2O^+ (aq), and $\Delta G_r > -2.55$ eV. The corresponding limit is $\Delta G > 3.01$ eV. Combining this limit with the upper limit $\Delta G = 3.58$ eV, one concludes that $\Delta G = 3.3 \pm 0.3$ eV.

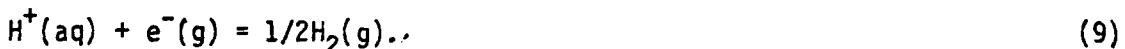
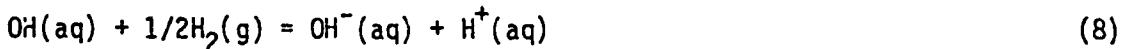
This result is consistent with the value $\Delta G = 3.4 \pm 0.5$ eV estimated in [15] from thermodynamic and kinetic data.

The changes of free energy for photoelectron emission by water are displayed in fig. 2. The corresponding standard reduction potentials are also shown. Reactions are written as oxidations (from left to right) for the scale of free energies and as reductions for the scale of standard potentials.

The least photon energy required for photolysis of water with production of hydrated electrons e^- (aq) can be estimated [15] from $E^0 = 3.3 \pm 0.3$ eV for $H_2O^+(aq)/H_2O(aq)$ and $E^0 = -2.77$ eV [16] for e^- (aq). Thus, a photon energy of at least $2.77 + 3.3 \pm 0.3 \approx 6.1 \pm 0.3$ eV is required to produce e^- (g). Formation of this species is indeed observed below 6.5 eV [16].

3. Photoelectron emission by hydroxide ion

The previously reported [3] threshold energy $E_t = 8.45$ eV of OH^- (aq) ion was confirmed in the present investigation. This result will be interpreted by the sequence of reactions:



The hydroxyl $OH(aq_*)$ produced by photoionization in (6) has initially the solvation configuration of $OH^-(aq)$ except for the loss of electronic polarization. Subsequent reorganization in (7) yields $OH(aq)$. The O-H interatomic distance can be taken as constant during this process since this distance is the same within 0.002 Å for $OH^-(g)$ and $OH(g)$ [17]. The loss of orientation polarization therefore is the sole contribution to ΔG_r for (7). The effect of OH^- on the surface potential of water is considerably smaller than that of Cl^- [13], and the corresponding uncertainty can be taken to be that of water, namely ± 0.1 V [11].

The value of ΔG_e deduced from (6) to (9) is given by eq. (1) where $\Delta G_e \approx E_t = 8.45$ eV and $\Delta G = 1.97$ eV [15]. One computes from (1) $\Delta G_r = 1.98 \pm 0.1$ eV. The changes of free energy and standard reduction potentials are shown in fig. 2 for photoelectron emission by hydroxide ion.

The ratio $\Delta G_r/\Delta G_s$ for the hydroxide ion will be determined to complement the results of Table 1. The solvation enthalpy of OH^- (aq) is -4.78 ± 0.25 eV [18]. This quantity can also be computed from a cycle of reactions involving the electron affinity (1.83 ± 0.04 eV [17]) of $\text{OH}(\text{g})$. The free energy ΔG_s for this cycle can also be calculated if the unknown entropy contribution is neglected for the process $\text{OH}(\text{g}) + \text{e}^-(\text{g}) = \text{OH}^-(\text{g})$. The entropy contribution to the solvation free energy ΔG_s of OH^- (aq) is estimated in this way at ca. 0.6 eV from the enthalpy and free energy calculations. Thus, $\Delta G_s = -4.78 + 0.6 \pm 0.25 \approx -4.2 \pm 0.3$ eV. Since $\Delta G_r = -1.98 \pm 0.1$ eV for OH^- (aq), one obtains $0.44 < \Delta G_r/\Delta G_s < 0.51$. This result is comparable to the more accurate ratios for the halides in Table 1.

4. Correlation between the threshold energies of water and hydroxide ion

The threshold energies of H_2O (aq) and OH^- (aq) will be correlated using fig. 3. The levels at -2.46 and -1.63 eV in this diagram correspond, respectively, to the free energies of formation of H_2O (aq) and OH^- (aq) [19]. These levels differ by $0.0592 \log 10^{-14} = -0.83$ eV. One has $\Delta G = 3.3 \pm 0.3$ eV, $\Delta G_H = 4.5$ eV, $\Delta G_r = -2.26 \pm 0.3$ eV for emission by H_2O (aq) (sec. 2). Likewise, $\Delta G = 1.97$ eV, $\Delta G_H = 4.5$ eV, $\Delta G_r = -1.98 \pm 0.1$ eV for emission by OH^- (aq) (sec. 3). One deduces from fig. 3,

$$\begin{aligned}
 \Delta G_e(\text{H}_2\text{O}) - \Delta G_e(\text{OH}^-) &= [\Delta G(\text{OH}^-) - \Delta G(\text{H}_2\text{O})] \\
 &+ [\Delta G(\text{H}_2\text{O}^+) - \Delta G(\text{OH})] + [\Delta G_r(\text{OH}) - \Delta G_r(\text{H}_2\text{O}^+)] \\
 &= (-1.63 + 2.46) + (0.84 - 0.34) + (-1.98 + 2.26) \\
 &= 0.83 + 0.50 + 0.28 \\
 &= 1.61 \text{ eV.}
 \end{aligned} \tag{10}$$

The symbol (aq) is deleted from all species in (10) to simplify the writing. The sum of the quantities between the first and second pairs of brackets in (10) represents the difference between the free energy changes $\Delta G = 3.3$ and 1.97 eV for emission by $H_2O(aq)$ and $OH^-(aq)$.

The threshold energies ($\Delta G_e \approx E_t$, sec. 2) of $H_2O(aq)$ and $OH^-(aq)$ are different, according to (10), for the following reasons: (i) The free energy of formation is less negative by 0.83 eV for $OH^-(aq)$ than for $H_2O(aq)$. This is the case because water is only slightly dissociated. (ii) The $OH(aq)$ radical does not protonate even in acid (1 M) solution [15], and therefore the free energy of formation of $H_2O^+(aq)$ must be more positive than that of $OH(aq)$. Decomposition of $H_2O^+(aq)$ into $OH(aq)$ and $H^+(aq)$ is favored. (iii) The reorganization free energies of $H_2O^+(aq)$ and $OH(aq)$ are not equal (but not very different).

Acknowledgment

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Table 1

Experimental and calculated $\Delta G_r/\Delta G_s$ ratios for the halides

	E_t^a (eV)	ΔG^b (eV)	$-\Delta G_r$ (eV)	$-\Delta G_s$ (eV)	$\Delta G_r/\Delta G_s$ (experimental)	$\Delta G_r/\Delta G_s^c$ (theory)
F^-	-	-	-	4.66	-	0.50
Cl^-	8.81	2.51	1.80	3.46	0.52	0.50
Br^-	8.05	1.98	1.57	3.18	0.49	0.49
I^-	7.19	1.32	1.37	2.81	0.49	0.48

a) With correction for absorption in water vapor and LiF window (sec. 1).

b) ΔG for X^- (aq) = X (aq) + e^- (g). ΔG includes $\Delta G_s = 0.06$ eV for X (g) = X (aq) [10].c) ΔG_s calculated from eq. (8) in [14]. $\Delta G_r = \Delta G_s - \Delta G_{ep}$, where ΔG_{ep} (< 0) is computed from the Born equation.

Captions to Figures

Fig. 1. Photoelectron emission spectrum of liquid water at 1.5°C (curve A) and plot of the yield (Y) to the power $n = 0.5$ vs. photon energy (line B). Plot for statistical F-test of the exponent n of the yield in inset. $F = R^2(N - 2)/(1 - R^2)$, where R is the correlation coefficient for least square fitting of line B and N the number of points.

Fig. 2. Free energy changes (vs. vacuum level of the electron) and standard reduction potentials for photoelectron emission by water and hydroxide ion. Uncertainties given in text.

Fig. 3. Free energies of formation and free energy changes for photoelectron emission by water and hydroxide ion. Symbol (aq) deleted from all species. e^- (g) represents the electron in the gas phase.

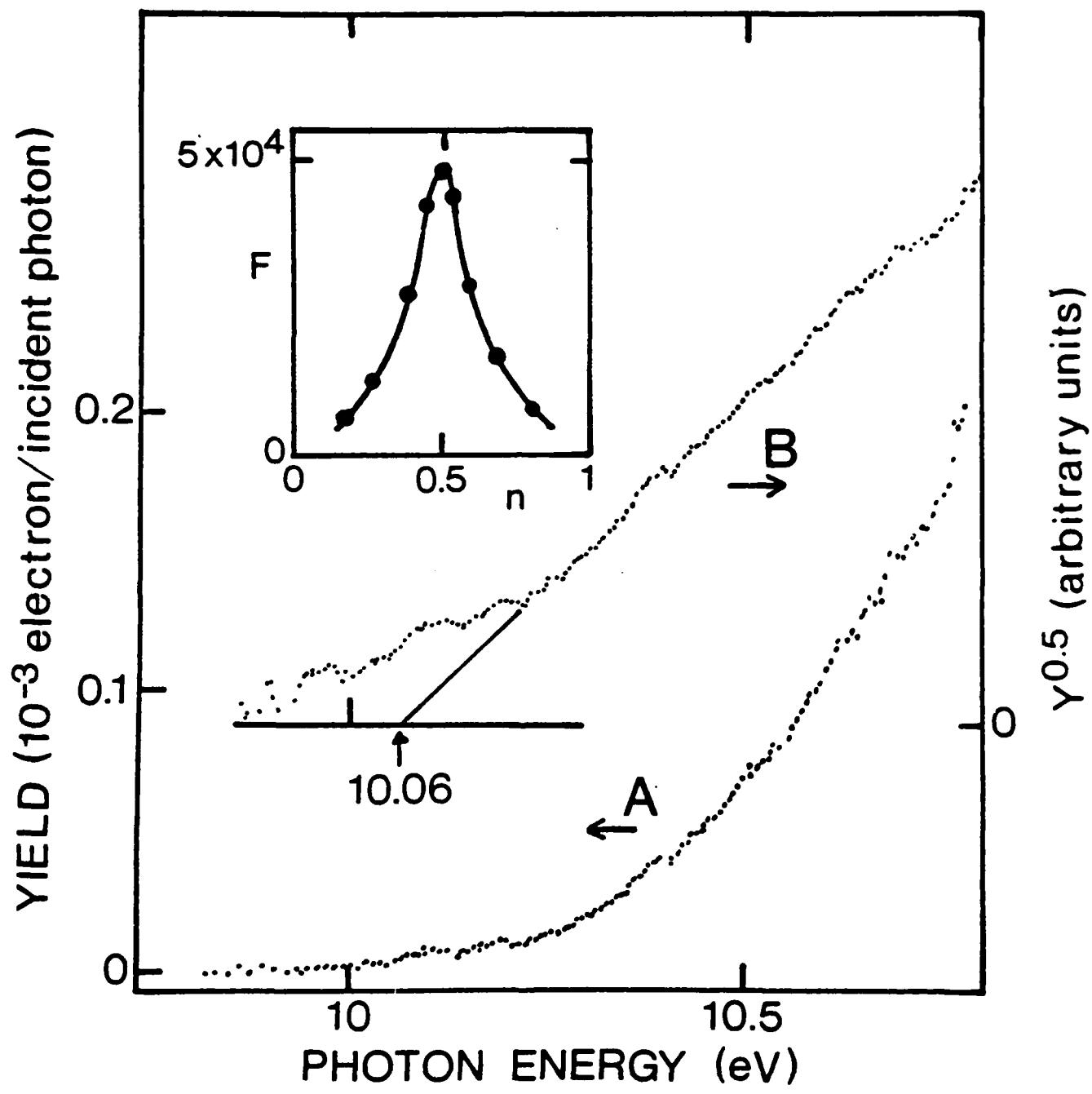


FIG. 1

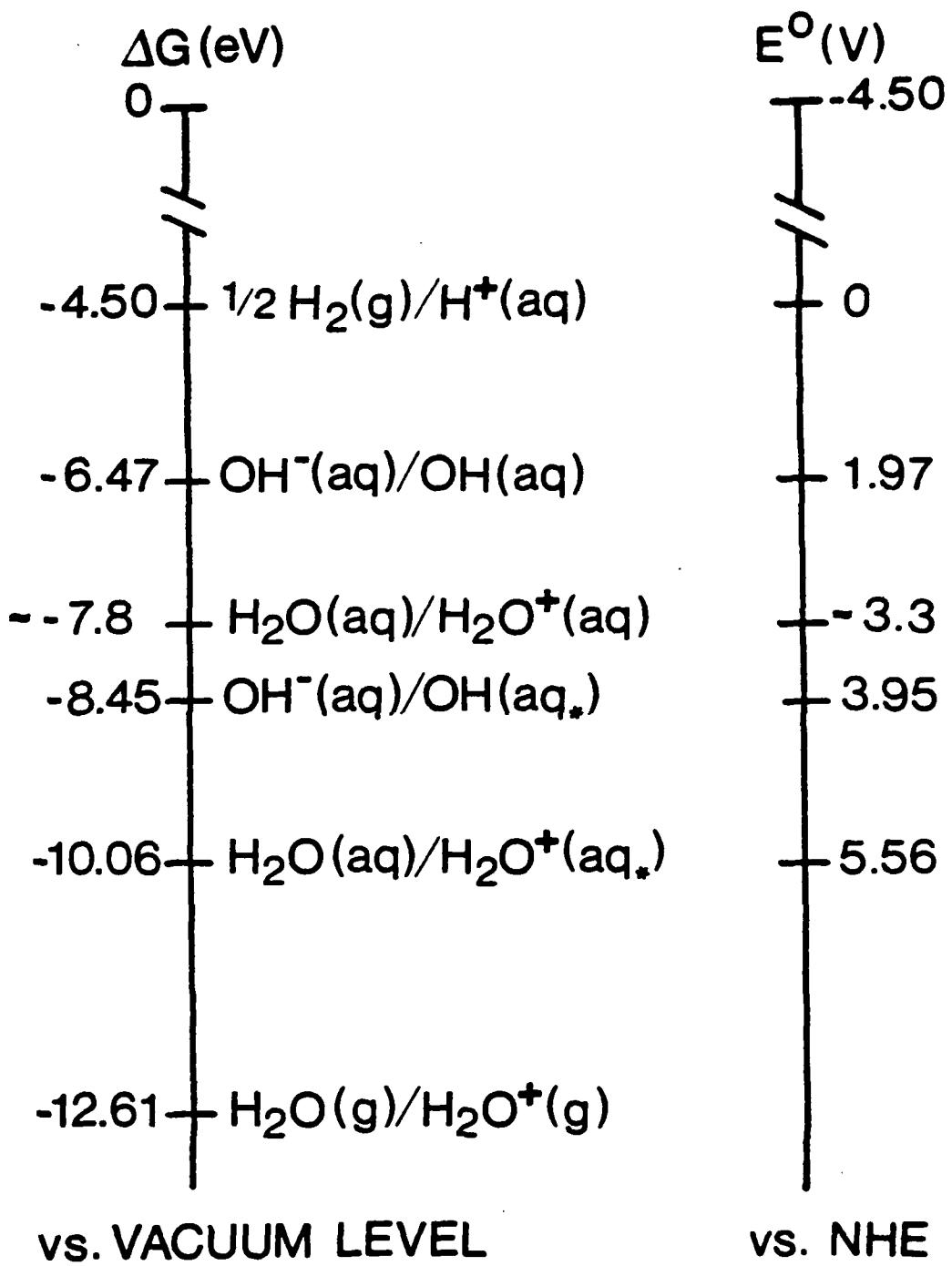


FIG. 2

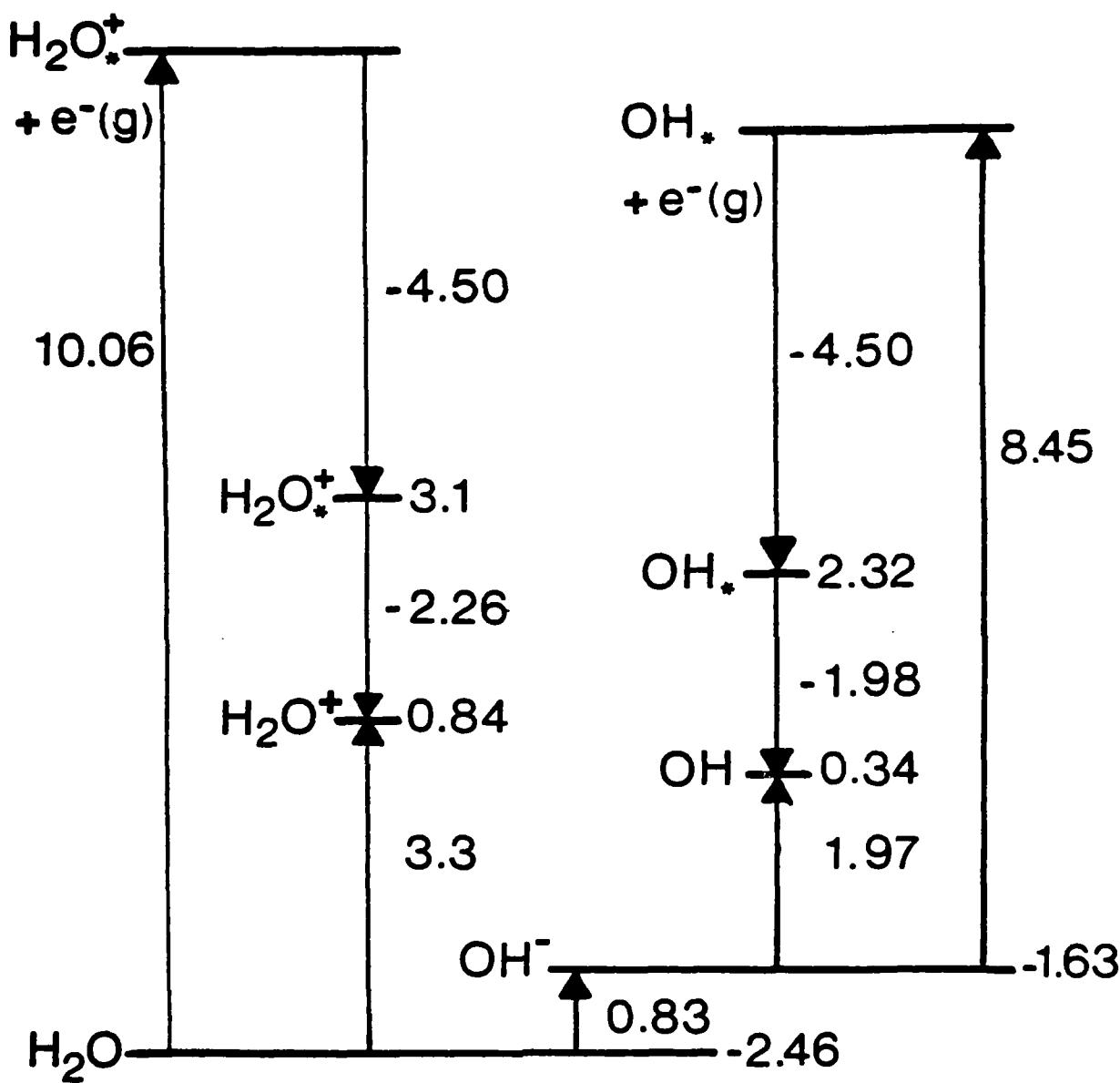


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